

**“A feasibility study of UV laser assisted 3D-atom probe analysis of AlGaIn/GaN HEMTs”**

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**Abstract:** Short summary of most important research results that explain why the work was done, what was accomplished, and how it pushed scientific frontiers or advanced the field.

Field evaporation of Air Force Research Laboratory AlGaIn/GaN high electron mobility transistors was attempted using a 3-D atom probe system with a 343nm UV wavelength laser. Previous attempts to analyze these samples using a Local Electrode Atom Probe system with a 532nm wavelength laser were unsuccessful. This work was completed to study (1) the factors (temperature, laser power, ambient, and sample orientation) affecting field evaporation of AlGaIn/GaN high electron mobility transistors and (2) using laser assisted field evaporation to measure the chemical composition of gate metal/AlGaIn interface region on the nano-scale in order to correlate changes in composition with electrical and physical degradation using site specific regions identified with infra-red spectroscopy, photoemission electron microscopy, and electron beam induced current analysis techniques. Additionally, previous studies using this 343nm UV wavelength system on ceramic oxide materials, Li ion battery powders, and (Ga,Mn)As thin films on GaAs substrates which exhibit poor electrical and thermal properties like the wide bandgap AlGaIn/GaN epilayers have been successfully. However, in this study every atom probe tip from the devices fractured before a successful ‘complete’ field evaporation consisting of the gate metal, interfacial layer, and AlGaIn/GaN epilayers occurred. This tip fracture issue is possibly due to the presence of a thick interfacial layer between the gate metal and AlGaIn epilayer. This interfacial layer could lower the adhesion strength between the gate and AlGaIn leading to the fracture and delamination observed during the laser assisted field evaporation due the magnitude of the electric field on the tip apex. However, it is noted that increasing temperature, lowering laser power, and introducing of a slight pressure of Ne gas helped improve parts of the field evaporation of the samples.

**Introduction:** Include a summary of specific aims of the research and describe the importance and ultimate goal of the work.

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The aim of this research is to determine the factors that influence the field evaporation of Air Force Research Laboratory (AFRL) high electron mobility transistors (HEMTs), and to successfully field evaporate the complete gate region (gate metal, interfacial layer and defects, and AlGaIn/GaN epilayers) of an AFRL HEMT using laser assisted 3-D atom probe (3DAP). Successful field evaporation of the complete region would permit a nano-scale chemical analysis comparison of pre- and post-stressed HEMTs. This is important because a relationship between the change in composition of the gate region and electrical characteristics of the HEMT could be obtained. From this change, defect formation mechanisms and reaction pathways can be proposed and tested. Once verified or rejected, the proposed mechanisms would provide insight into the failure mechanisms of AlGaIn/GaN HEMTs. Ultimately, these mechanisms would be added to the Florida Object Oriented Reliability Simulator (FLOORS) being developed at the University of Florida by Dr. Mark Law to help predict device reliability and lifetimes which would ultimately help improve device design and reliability of AlGaIn/GaN HEMTs by making simulations more accurate.

**Experiment:** Description of the experiment(s)/theory and equipment or analyses..

In order to vary the amount of composition change between HEMTs, three different gate length devices were used consisting of 100nm, 170nm, and 1 $\mu$ m. By changing the gate lengths while keeping the stress conditions the same between the HEMTs, the internal electric fields will be different magnitudes. Because of the piezoelectric nature of the materials, the difference in electric field magnitude would result in each device experiencing a different amount of mechanical stress and degradation. The characteristic electrical curves of pre-stressed AlGaIn/GaN HEMTs were measured using an HP 4156C semiconductor parameter analyzer. Following baseline measurements, the devices were stressed using off-state high reverse gate bias conditions. For the stress conditions, the  $V_{DS}$  was constant at 5V while the  $V_G$  was stepped from -10V to -42V at -1V/min. After the applied stress, the characteristic electrical curves were measured again for each device to provide comparison between the pre- and post-stressed HEMTs.

Following the applied electrical stress and measurement, the HEMTs were shipped to AFRL where infra-red spectroscopy (IR), photoemission electron microscopy (PE), and electron beam induced current (EBIC) analysis were performed. These analysis techniques each provided a map of the entire gate region of the devices indicating the location of hot spots along the gate width. Using these maps, site specific focus ion beam (FIB) sectioning was used to fabricate samples for 3DAP analysis from both hot spot and non-hot spot areas for comparison.

However, in order to find the optimal field evaporation conditions and reduce the likelihood of atom probe sample tip fracture, the sample temperature, laser power, and ambient were systematically varied. The temperature ranged from 22K to 60K, laser energy was varied between 0.4mW to 10mW, and the ambient consisted of ultra high vacuum conditions with the occasional addition of a slight pressure of Ne which was added to help reduce the electric field at the sample tip. Additionally, while keeping the temperature, laser power, and ambient at the same conditions, the orientation of the gate

region with respect to the tip apex was changed in order to determine the effect of sample orientation on the quality of the field evaporation.

The atom probe tips were fabricated using an in-house system in the Magnetic Materials Unit (MMU) at the National Institute for Materials Science (NIMS). This procedure involved electropolishing W wires until the diameter approached 500nm. Then the AFRL devices were microsampled using a Hitachi FB-2100. During this process, the W wires are milled until the tip was flat and approximately 2  $\mu\text{m}$  in diameter so that microsampled AFRL HEMTs could be mounted onto the W wire using the in-house wire holder system. The W wire and sample were then transfer to a Carl Zeiss Cross Beam 1540 EsB FIB and were subjected to annular milling until the tip apex was below 100nm and within 20 to 50nm of the region of interest. After the milling was completed, the finished atom probe tips were transferred into the laser assisted wide angle 3DAP to await field evaporation and analysis. Following evaporation, the collected data was analyzed using PoSAP or IVAS software packages.

**Results and Discussion:** Describe significant experimental and/or theoretical research advances or findings and their significance to the field and what work may be performed in the future as a follow on project. Fellow researchers will be interested to know what impact this research has on your particular field of science.

Many factors affected the quality of the field evaporation of the AFRL AlGaIn/GaN HEMTs. Increasing specimen temperature increased the evaporation rate, but it did not reduce the frequency of the sample tip fracture. Decreasing laser power increased the time until specimen fracture, but it also did not prevent fracture. However, if the laser power was too large, the laser would damage the sample preventing evaporation. Concurrent scanning electron microscopy (SEM) during FIB atom probe tip fabrication reveals in Fig. 1(a) a specimen tip after complete fabrication and Fig. 1(b) a sample after fracture when using a laser power of 10mW. By comparing the thickness of the specimen (since all atom probe tips in this study are approximately the same geometry), the distance from the tip to the sample fracture can be estimated. Fig. 1(a) shows that the tip has a diameter of approximately 1 $\mu\text{m}$  about 10 $\mu\text{m}$  from the apex of the tip. However, in Fig. 1(b), the diameter of the tip at the fracture surface is approximately 2 to 4 $\mu\text{m}$  indicating that the specimen fracture occurred more than 10 $\mu\text{m}$  away from tip and area of interest. Thus, lowering the laser power prevents the catastrophic fracturing of tips from AlGaIn/GaN HEMTs. Additionally, it is noted that adding a slight amount of He gas to the vacuum during evaporation also reduced the time until fracture for the specimen. The addition of He gas would reduce the magnitude of the electric field at the tip of the specimen. Thus, if there is a weak interface in the specimen, it may not fracture as quickly.

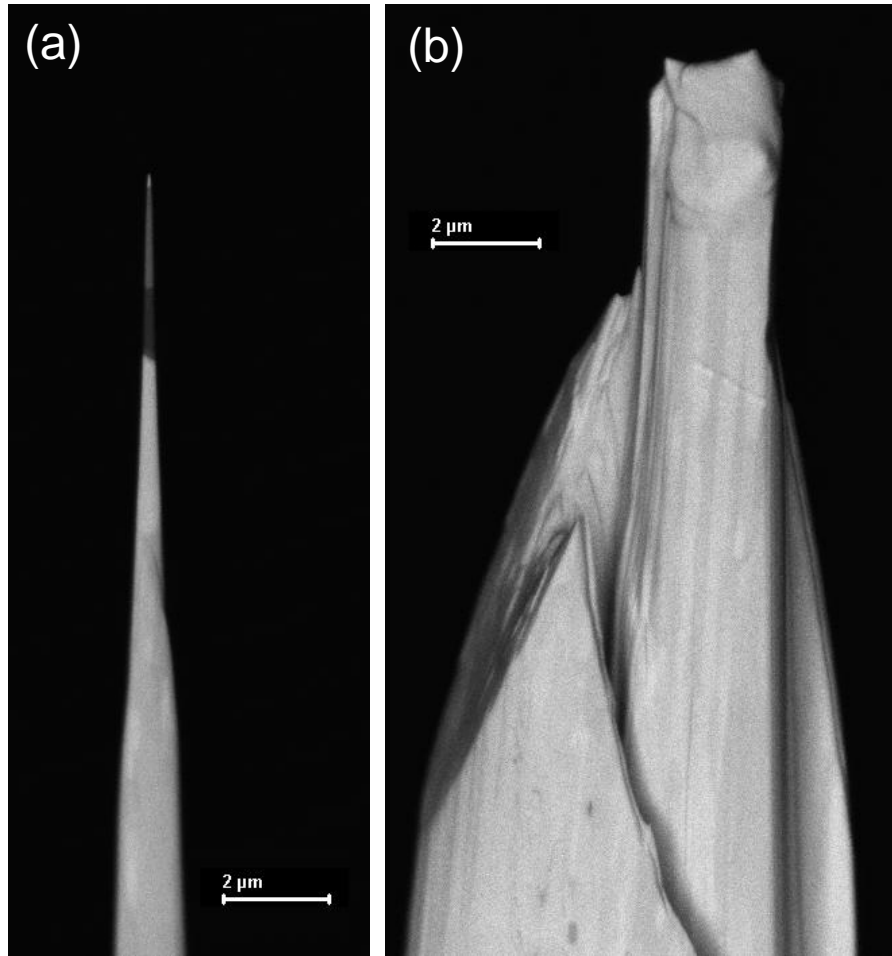


Fig. 1: (a) A backscattered SEM micrograph showing a freshly completed atom probe tip of an AlGaIn/GaN HEMT and (b) a backscatter SEM micrograph showing an atom probe tip after laser assisted field evaporation utilizing a laser power of 10mW.

Furthermore, the orientation of the specimen with respect to the tip apex was studied. Because fracture predominately occurred at the gate/AlGaIn interface, different sample orientations were used to try to prevent fracture. By changing the orientation between the interfacial layer and the tip apex, the different layers (gate metal, interfacial layer, and AlGaIn/GaN epilayers) are exposed at different and varying times during the evaporation. This affects the evaporation of the tip due to the change in electric field distribution at the tip apex due to the change in tip apex composition. For the sample orientation comparison, a transmission electron microscopy (TEM) micrograph of a representative cross section of a 1  $\mu\text{m}$  gate length AFRL AlGaIn/GaN HEMT is presented in Fig. 2(a). Here, high-angle annular dark-field scanning TEM (HAADF-STEM) was used. The contrast mechanism correlates to the atomic number where brighter features correspond to areas of greater atomic number. In Fig. 2(a) the two bright layers of the gate are the Au and Ni layers while the two darker layers below them are the AlGaIn and GaN epilayers. Additionally, Fig. 2(a) shows the directions of analysis for the four different orientations studied. The labels next to the direction of analysis in Fig. 2(a) correspond to Fig. 2(b-e) respectively.

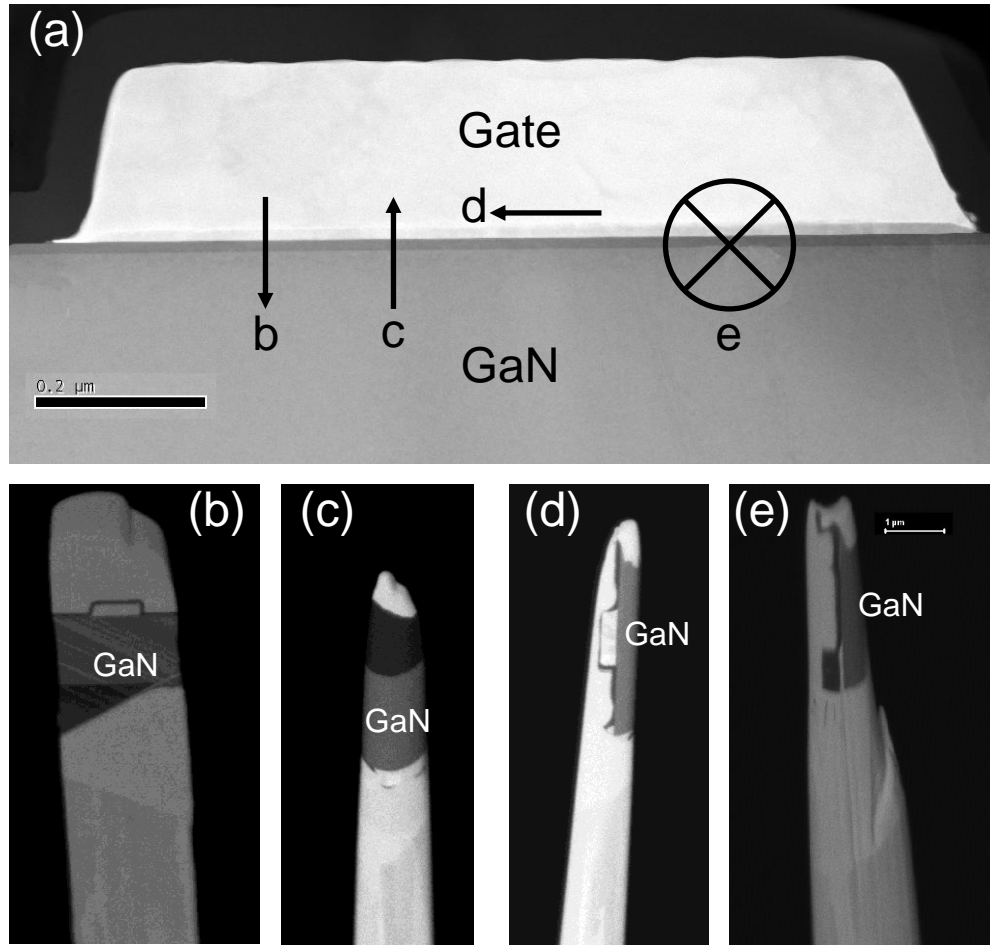


Fig. 1: (a) A HAADF-STEM micrograph showing the cross section of a AFRL AlGaIn/GaN HEMT including the gate metal and AlGaIn and GaN epilayers. The analysis direction for the different sample orientations is indicated. (b) A backscatter SEM micrograph of a pre-sharpened standard top-down oriented atom probe tip. (c) A backscatter SEM micrograph of a pre-sharpened reverse top-down oriented atom probe tip. (d) A backscatter SEM micrograph of a pre-sharpened 90° rotated with respect to the HEMT cross section atom probe tip. (e) A backscatter SEM micrograph of a pre-sharpened 90° rotated out of the plane of the cross section atom probe tip.

The standard top-down orientation where the sample tip is fabricated so that it matches the TEM cross section is shown in a pre-sharpened state in Fig. 2(b). Here, the gate region is mounted above the AlGaIn/GaN epilayers and evaporation begins in the metal and progresses into the epilayers. The reverse top-down orientation is shown in Fig. 2(c) in a pre-sharpened state and is the opposite of the standard orientation. Here, the epilayers are mounted above the gate so evaporation begins in the GaN and progresses into the gate metal. Additionally, it is noted that in some samples additional metal was deposited over the gate in order to increase the distance between the gate and the weld between the microsampled HEMT and W wire. The next orientation consists of a 90° rotation of the gate/AlGaIn interface with respect to the cross section of the HEMT. In this orientation as shown in Fig. 2(d) evaporation occurs at the gate, interfacial layer, and AlGaIn/GaN epilayers at the same time. The last orientation again consists of a 90° rotation of the gate/AlGaIn interface but this time out of the plane of the cross section of the HEMT as shown in Fig. 2(e). Once again evaporation occurs at the gate, interfacial

layer, and AlGaIn/GaN epilayers at the same time, but in this case evaporation could continue down the gate width of the device while in the previous orientation it would only be possible for the 1  $\mu$ m across the gate length. From these four orientations, the standard top-down orientation provided the best field evaporation before tip fracture. The reverse top-down orientation exhibited poor evaporation and fractured. Additionally, the two 90° rotation orientations had the worst results with fractures occurring immediately upon the initiation of field evaporation. These results indicate that including the interfacial layer in the immediate field evaporation resulted in early fracture of the tip, and may indicate that the interfacial layer is a weak feature in the sample that contribute to the samples constantly fracturing.

While there was no ‘complete’ field evaporated specimen obtained, the best atom probe run resulted from the standard top-down orientation at 40K, 0.5 mW, and a small amount of He gas added. This run showed part of the AlGaIn epilayer and continued down into the GaN epilayer which is shown in Fig. 3. Here, a slightly amount of O is seen at the top of the AlGaIn epilayer. This could be the bottom of part of the interfacial layer although no other ions were detected in this region, or it is possibly some kind of contamination in the sample or from a fracture in the gate metal above the AlGaIn. The majority of the other fractured tips only resulted in the evaporation of the Au layer of the gate followed by the beginning of the Ni evaporation, and then concluded with a sample tip fracture and evaporation of only GaN. Fig. 3 shows the only run to contain part of the AlGaIn epilayer. Thus, the interfacial layer between the gate and AlGaIn epilayer and the top part of the AlGaIn layer were not analyzed.

Due to the fracture issues during the laser assisted field evaporation of the AFRL AlGaIn/GaN HEMTs and the observation that the interfacial layer may play a role in the fractures, a collaboration with Dr. Curtis Taylor at the University of Florida has been established. This on-going work will characterize the adhesion strength of the gate metal to the AlGaIn epilayer by comparing the force required to shear the gate off the surface of different AlGaIn/GaN HEMTs. This work will permit a comparison between the sheer force required between non-AFRL HEMTs which were previously ‘completely’ field evaporated on different atom probe tomography systems and the AFRL devices that have not had a successful ‘complete’ evaporation on any atom probe tomography system. Should a difference between the sets of HEMTs exist, this may show that the native interfacial layer present in the AFRL AlGaIn/GaN HEMTs could be the feature preventing 3DAP analysis.



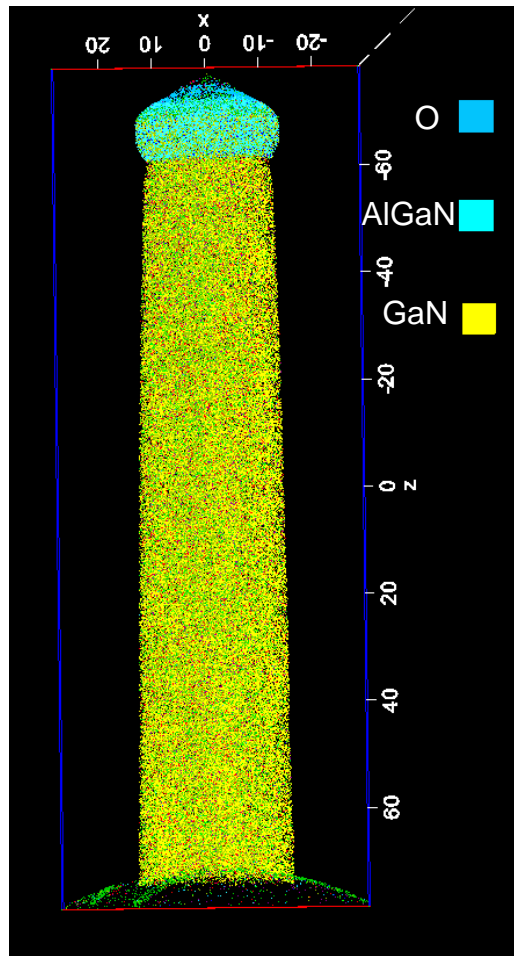


Fig. 3: (a) Partial atom probe tomography reconstruction showing part of the AlGaIn epilayer and the GaN layer below it. Additionally, there is layer of O above the AlGaIn epilayer which may be part of the interfacial layer between the gate metal and the AlGaIn or contamination from the metal fracturing above the AlGaIn.

**List of Publications and Significant Collaborations that resulted from your AOARD supported project:** In standard format showing authors, title, journal, issue, pages, and date, for each category list the following:

- a) papers published in peer-reviewed journals,
- b) papers published in peer-reviewed conference proceedings,
- c) papers published in non-peer-reviewed journals and conference proceedings,
- d) conference presentations without papers,
- e) manuscripts submitted but not yet published, and
- f) provide a list any interactions with industry or with Air Force Research Laboratory scientists or significant collaborations that resulted from this work.

f) This research grant had multiple interactions with scientists at the Air Force Research Laboratory in Dayton, Ohio, primarily with Dr. Eric Heller and Steve Tetlak. Dr. Eric Heller provided the IR and PE analysis on stressed HEMTs, and Steve Tetlak provided the EBIC analysis. Additionally, Steve Tetlak grounded the samples to metal substrates and covered them with a small layer of Au which helped reduce the charging the samples experienced in the FIBs. This stopped sample drift in the FIBs allowing the successful fabrication of site-specific atom probe tips.

Furthermore, there are many other significant collaborations that have resulted from this work. Dr. Fan Ren and Dr. Stephen Pearton at the University of Florida stressed the HEMTs using off-state high reverse gate bias conditions and recorded the pre- and post-stressed characteristic electrical curves. Dr. Gijs Bosman at the University of Florida has provided trapping analysis from the pre- and post-stressed HEMTs. Dr. Kevin Jones at the University of Florida has provided TEM micrographs of the pre- and post-stressed HEMTs. Lastly, Dr. Curtis Taylor of the University of Florida has recently begun a collaborative effort to help study the effect of the interfacial layer thickness on the gate adhesion strength.

**Attachments:** Publications a), b) and c) listed above if possible.

**DD882:** As a separate document, please complete and sign the inventions disclosure form.

**Important Note:** If the work has been adequately described in refereed publications, submit an abstract as described above and refer the reader to your above List of Publications for details. If a full report needs to be written, then submission of a final report that is very similar to a full length journal article will be sufficient in most cases. This document may be as long or as short as needed to give a fair account of the work performed during the period of performance. There will be variations depending on the scope of the work. As such, there is no length or formatting constraints for the final report. Keep in mind the amount of funding you received relative to the amount of effort you put into the report. For example, do not submit a \$300k report for \$50k worth of funding; likewise, do not submit a \$50k report for \$300k worth of funding. Include as many charts and figures as required to explain the work.